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DEVELOPMENT OF A FLOWSHEET FOR THE TREATMENT OF CHLORIDE LEACH LIQUOR: SELECTION OF A SOLVENT EXTRACTION REAGENTS.

PROGRESS REPORT NO. 2

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by

G.M. RITCEY and B.H. LUCAS

INTRODUCTION

This progress report is the second in a series describing the research pertaining to the selection of solvent extraction reagents in the development of a flowsheet for the purification of chloride leach liquors. In the previous work[1], it was considered that the leach solutions arising from the chlorination of the complex Pb/Zn sulphide concentrate and subsequent leaching would contain at least 0.5 g Fe/ℓ, and that the leach solution would be at a pH of 1.8. The results of screening tests on various extractants for the feasibility of treating a solution containing in g/ℓ: 25 Zn, 0.5 Cu, 0.5 Fe^{3+}, at pH 1.8 were as follows:

(1) Alamine 336 could be used for extraction and removal of ferric iron and recovery as ferric chloride prior to recovery of other metals;

(2) SME 529 could be used for extraction of copper followed by copper sulphate stripping;

(3) Alamine 336 could be used to extract and recover zinc as zinc chloride;

(4) D2EHPA could be used for extraction of zinc, as an alternative, and recovery of zinc sulphate by stripping with H_2SO_4.

The subsequent work was concerned with the examination and comparison of other possible extractants for copper and the discrimination over other metals such as zinc and small amounts of iron. The effect of the total chloride content in the leach liquor (as NaCl) was also studied for its effect on extraction and discrimination. Screening tests were carried out for the extraction of lead. Also, additional work on the extraction of zinc and discrimination over other metals was performed.

PROCEDURE

Apart from some initial tests for copper on synthetic solutions, the majority of the tests were conducted on actual roast-leach liquor where the metal concentrations were adjusted to a desired value. The tests were all conducted in separatory funnels, where the extraction parameters such as equilibrium pH, contact time, chloride concentration, were varied. All extractants in the copper and lead series were 10 v/o. The TBP extractant for Zn was varied in concentration up to 100 v/o. Prior to extraction, all solvents were pre-equilibrated as required unless otherwise noted. The solution composition was approximately the same as the synthetic solution used formerly,[1] except for the addition of 0.4 g Pb/ℓ.

The concurrent work on the chlorination-oxidation-leach indicated that by control of the operating conditions, the resulting leach liquor would contain <0.1 g Fe/ℓ. Therefore, in the work reported here, the iron content was ignored with respect to its prior extraction, as it may not be necessary, in actual practice, to provide a separate removal circuit.

With respect to iron, it was noted that the SME 529 extractant of Shell contained an appreciable iron content when received from the manufacturer. It was only after many tests had been performed that an analysis of the extractant showed 22 g Fe/ℓ in the stock solution, or one-tenth that amount in the 10% extractants used in the tests. Hence such an amount would have to be removed prior to its use in a plant operation. Prior stripping with HCl proved effective in this regard, as compared to negligible removal if stripped with NaCl solution.

RESULTS

Copper Extraction

Screening of Extractants

Effect of Contact Time and Chloride Concentration on Extraction and Selectivity Using Selected Extractants

Tests were conducted on a synthetic chloride leach solution, having the following analyses, in g/ℓ: 0.54 Cu, 0.52 Fe, 25.3 Zn in 1M NaCl. The A/O ratio was 1 in tests conducted on 10 v/o solutions of LIX 70, Kelex 100 and Shell 529. The LIX 70 and Shell 529 were dissolved in Shell 140 kerosene, while the Kelex 100 with 10% isodecanol, was dissolved in

Solvesso 150. All extractants were pre-equilibrated with 150 g/ℓ HCl, O/A of 5, and water washed before using. The kinetic study was over the range 10 seconds to 5 minutes, at an equilibrium pH of 1.5. The results indicated that SME 529 was the best extractant and most selective of those tested for discrimination over Fe and Zn. A contact time of 2 minutes appeared optimum for maximum extraction and separation from Fe and Zn.

Subsequent tests using the same extractants were duplicated on actual leach liquor obtained by the chlorination-leach procedure. The extraction at an equilibrium pH 1.5 and 2.0 were compared, and the chloride concentration was varied by the addition of NaCl, in the range 0-5M NaCl. The feed analyzed, in g/ℓ: 0.5 Cu, 0.6 Fe, 24.0 Zn. The results indicated all three extractants were capable of the selective extraction of copper, but the SME 529 was the most selective over Zn and Fe. Kinetics had little effect in the low chloride solution (0 NaCl addition), both at equilibrium pH 1.5 and 2.0, and 2 minutes appeared sufficient for copper extraction. However, as the NaCl concentration was increased from 0-5M, the Fe loading was increased with retention time. Both LIX 70 and SME 529 loaded similar amounts of iron, compared to Kelex 100 which loaded considerably more iron. For example, at 1M NaCl, SME and LIX 70 co-extracted approximately 10% of the Fe and at 5M NaCl about 15%, compared to 25% and 85% at 1M and 5M NaCl, respectively, for Kelex. Negligible amounts of Zn were co-extracted with the SME 529 compared to higher amounts for the LIX 70 and

Kelex 100. For example, at 1M and 5M NaCl concentrations, the loaded SME solvent contained 0.0002 g Zn/ℓ and 0.003 g Zn/ℓ respectively, compared to 0.0009 and 0.002 for LIX 70 and 0.0002 and 0.84 for Kelex. A screening of the results are shown in Table 1.

The results in Table 1 also indicate the following:

(1) The Cu/Fe discrimination is approximately equal for the three reagents, under the conditions tested. There was no apparent effect on discrimination due to the pH or NaCl concentration.

(2) The Cu/Zn discrimination was similar for LIX 70 and SME 529. Kelex 100 was the inferior of the three extractants. At low pH 1.5 increasing the NaCl concentration from 0 to 1M resulted in a dramatic increase in the copper selectivity over zinc compared to the tests at pH 2.0. Very little change occurred with increase in NaCl concentration from 2 to 5M.

From the screening tests SME 529 was selected for further work on copper extraction, and those results are detailed later in this section. However, during that period of investigation on SME 529, other chelating extractants were considered possible for the copper circuit. These were the new Acorga* reagents. Therefore screening tests were conducted on two Acorga extractants, together with SME 529 for comparison and, in addition, LIX 64N and LIX 65N to cover a better range. The extractants were as 10% solution in kerosene, pre-equilibrated with 150 g HCl/ℓ at A/O 5 followed by a water wash at A/O 1.

*produced by Imperial Chemical Industries Ltd.

TABLE 1

Screening of Extractants

Effect of Contact Time, Chloride Concentration and pH

Extractant	Equil. pH	NaCl Added M	Contact Time Sec.	Solvent Loading (g/ℓ)			Separation	
				Cu	Fe	Zn	Cu/Fe	Cu/Zn
LIX 70	1.5	0	10	.48	.01			
			30	.50	.01	.02		
			120	.51	.01	.02		
			300	.51	.036	.06	14	8
Kelex 100			10	.50	.01			
			30	.51	.02			
			120	.51	.04			
			300	.51	.08	.028	6	18
SME 529			10	.48	-			
			30	.50	.03			
			120	.48	.03			
			300	.49	.035	.0006	14	816
LIX 70	2.0	0	10	.48	.02			
			30	.48	.09			
			120	.48	.06			
			300	.48	.06	.0008	8	600
Kelex 100			10	.47	.07			
			30	.48	-			
			120	.48	-			
			300	.48	.05	.024	10	20
SME 529			10	.47	.01			
			30	.47	-			
			120	.47	-			
			300	.49	.06	.0008	8	612

TABLE 1

Screening of Extractants

Effect of Contact Time, Chloride Concentration and pH

Extractant	Equil. pH	NaCl Added M	Contact Time Sec.	Solvent Loading (g/ℓ)			Separation	
				Cu	Fe	Zn	Cu/Fe	Cu/Zn
LIX 70	1.5	1.0	10	.50	.05			
			30	.50	.06			
			120	.50	.03			
			300	.50	.02	.0009	25	555
Kelex 100			10	.50	.09			
			30	.50	.10			
			120	.50	.13			
			300	.50	.22	.0002	2	2500
SME 529			10	.49	.10			
			30	.49	.09			
			120	.48	.08			
			300	.47	.06	.0002	8	2350
LIX 70	2.0	1.0	10	.50	.06			
			30	.50	.05			
			120	.50	.05			
			300	.50	.05	.0003	10	1666
Kelex 100			10	.50	.05			
			30	.50	.08			
			120	.50	.11			
			300	.49	.19	.079	3	6
SME 529			10	.48	.03			
			30	.49	.04			
			120	.49	.05			
			300	.46	.05	.001	9	460

TABLE 1

Screening of Extractants

Effect of Contact Time, Chloride Concentration and pH

Extractant	Equil. pH	NaCl Added M	Contact Time Sec.	Solvent Loading (g/ℓ)			Separation	
				Cu	Fe	Zn	Cu/Fe	Cu/Zn
LIX 70	1.5	3.0	10	.50	.10			
			30	.50	.16			
			120	.50	.13			
			300	.49	.21	.0008	2	612
Kelex 100			10	.50	.22			
			30	.50	.44			
			120	.50	.57			
			300	.50	.16	.35	3	1
SME 529			10	.50	.09			
			30	.50	.14			
			120	.50	.16			
			300	.49	.58	.002	1	245
LIX 70	2.0	3.0	10	.51	.09			
			30	.51	.17			
			120	.51				
			300	.51	.21	.0008	2	638
Kelex 100			10		Ppt'n in aqueous. Emulsion in organic No samples taken			
			30					
			120					
			300					
SME 529			10	.51	.10			
			30	.51	.19			
			120	.51				
			300	.51	.21	.0007	2	729

TABLE 1

Screening of Extractants

Effect of Contact Time, Chloride Concentration and pH

Extractant	Equil. pH	NaCl Added M	Contact Time Sec.	Solvent Loading (g/ℓ)			Separation	
				Cu	Fe	Zn	Cu/Fe	Cu/Zn
LIX 70	1.5	5.0	10	.45	.06			
			30	.45	.10			
			120	.44	.10			
			300	.44	.11	.002	4	220
Kelex 100			10	.45	.45			
			30	.45	.45			
			120	.45	.45			
			300	.45	.45	.84	1	0.5
SME 529			10	.43	.07			
			30	.43	.08			
			120	.44	.08			
			300	.43	.08	.003	5	143
LIX 70	2.0	5.0	10	.45	.05	Cloudy		
			30	.45	.12	Raff.		
			120	.45				
			300	.44	.04	.0008 Cloudy Raff.	11	550
Kelex 100			10	.45	.43			
			30	.45	.43			
			120	.45	.43			
			300	.44	.45	.029	1	15
SME 529			10	.43	.02			
			30	.43	.04			
			120	.43	.07			
			300	.43	.11	.003	4	143

The feed to extraction contained, in g/ℓ: 29.6 Zn, 0.41 Cu, 0.40 Pb, 0.0003 Fe. Extraction was varied over a pH 1-5 range, NaCl addition of 0-3M, and the retention time was 2 minutes at a phase ratio A/O of 7. The results, given in Tables 2 and 3, indicate that the Acorga P5100 is the best extractant for copper, followed very closely by Acorga P5300. However, although P5100 was the most selective over Fe and Pb, the P5300 was preferable for separation of Zn. There was a definite trend towards a decrease in metals selectivity with either of the two extractants with increase in pH or NaCl concentration at 0 and 2M NaCl. At the lower pH values, LIX 65N was the next best extractant, while at pH 3-5, the SME 529 was the next best to the Acorga reagents. With all the reagents, increase in pH resulted in an increase in the extraction of copper. Also, a slight decrease in copper extraction resulted with increase in the NaCl concentration. With 3M NaCl, Acorga P5300 gave the best selectivity of Cu/Zn, Cu/Fe, especially at pH 4, although copper extraction was slightly lower compared to P5100 and SME 529.

The above tests indicated that the Acorga reagents have more promise as copper extractants than the other reagents tested. Further work is required to determine kinetic effects, equilibrium data as well as the stripping properties.

Equilibrium Data for Copper Extraction Using SME 529

Bench-scale equilibrium shake-out tests were conducted on a similar feed leach liquor as above, except that 0.5 g Pb/ℓ was added to the solution. NaCl was added to a 3M concentration. The 10% SME 529 in Shell 140 was pre-equilibrated with 150 g HCl/ℓ

TABLE 2

Screening of Extractants

Effect of Equilibrium pH and Chloride Concentration on Selectivity

Extractant	Equil. pH	NaCl (M)	Selectivity (SF)			Cu Ext'n (%)
			Cu/Zn	Cu/Fe	Cu/Pb	
LIX 64N	1	0	6.1	20.3	30.5	2.1
	2		1150	460	230	16.0
	3		1275	510	255	17.7
	4		842	295	295	20.6
LIX 65N	1		950	190	190	13.2
	2		1510	755	755	52.6
	3		149	1115	1115	77.7
	4		518	1295	1295	90.2
SME 529	1		371	52	260	9.1
	2		625	179	625	43.6
	3		1290	322	1290	89.9
	4		1365	303	2730	95.1
Acorga P5100	1		2928	1025	2050	71.4
	2		568	1420	2840	99.0
	3		238	1430	1430	99.7
	4		126	967	1450	100
P5300	1		2200	550	550	38.3
	2		3012	602	2410	84.0
	3		870	1305	1305	90.9
	4		1400	1400	2800	97.6
LIX 64N	1	1.0	122	30	30	2.1
	2		550	110	440	15.3
	3		656	590	295	20.6
	4		580	290	580	20.2
	5		1066	213	320	22.3
LIX 65N	1		1100	330	330	11.5
	2		163	655	655	45.6
	3		264	168	925	64.5
	4		883	662	1325	92.3
	5		412	1235	1235	86.1
SME 529	1		540	45	270	9.4
	2		1783	153	535	37.3
	3		2630	329	1325	91.6
	4		937	351	1405	97.9
	5		367	357	1285	89.5

TABLE 2

Screening of Extractants

Effect of Equilibrium pH and Chloride Concentration on Selectivity

Extractant	Equil. pH	NaCl (M)	Selectivity (SF)			Cu Ext'n (%)
			Cu/Zn	Cu/Fe	Cu/Pb	
Acorga P5100	1	1.0	3133	940	940	65.5
	2		394	920	1380	96.2
	3		186	697	1395	97.2
	4		150	947	1420	99.0
	5		68	1320	1320	92.0
Acorga P5300	1		2500	500	500	34.8
	2		2467	171	1110	77.4
	3		2660	332	1330	92.7
	4		1290	516	1290	90.0
	5		690	153	1380	96.2
LIX 64N	1	3.0	850	170	170	6.2
	2		1400	280	560	11.5
	3		1933	290	580	21.3
	4		1575	630	630	23.1
LIX 65N	1		2500	375	938	27.5
	2		2040	1020	2040	74.7
	3		1295	1295	2590	94.9
	4		633	1266	2811	92.7
SME 529	1		1300	93	650	23.8
	2		518	207	2070	75.8
	3		663	265	2650	97.1
	4		518	288	2590	94.9
Acorga P5100	1		588	2350	2350	86.1
	2		867	1300	2600	95.2
	3		422	1265	2530	92.7
	4		394	1380	2760	100.0
Acorga P5300	1		1550	1550	1550	56.8
	2		1180	2360	2360	86.5
	3		2967	2670	2670	97.8
	4		1190	2380	2380	87.2

TABLE 3

Screening of Extractants

Effect of Equilibrium pH and Chloride Concentration on Copper Extraction

Extractant		% Cu Extracted			
		Equilibrium pH			
		1	2	3	4
0 NaCl	P5100	71.4	99.0	99.7	100.0
	P5300	38.3	84.0	90.9	97.6
	SME 529	9.1	43.6	89.9	95.1
	65N	13.2	52.6	77.7	90.2
	64N	2.1	16.0	17.7	20.6
0 NaCl	P5100	65.5	96.2	97.2	99.0
	P5300	34.8	77.4	92.7	90.0
	SME 529	9.4	37.3	91.6	97.9
	65N	11.5	45.6	64.5	92.3
	64N	2.1	15.3	20.6	20.2
0 NaCl	P5100	86.1	95.2	92.7	100.0
	P5300	56.8	86.5	97.8	87.2
	SME 529	23.8	75.8	97.1	94.9
	65N	27.5	74.7	94.9	92.7
	64N	6.2	11.5	21.3	23.1

at O/A 5 and water-washed prior to the tests. Contact time of the tests was 1 minute at an equilibrium pH 1.5. The feed analyzed, in g/ℓ: 0.48 Cu, 0.48 Fe, 25.6 Zn, 0.46 Pb. The equilibrium data are shown in Table 4, and in Figure 1 is shown the equilibrium curve for copper extraction. According to the calculated stages of the McCabe-Thiele construction in Figure 1, five stages would be required to achieve a raffinate of about 0.005 g Cu/ℓ.

TABLE 4

Equilibrium Data for SME 529

A/O Ratio	Equilibrium Analyses g/ℓ							
	Cu		Fe		Zn		Pb	
	Raff	Org	Raff	Org	Raff	Org	Raff	Org
1/2	.013	.22	.41	.029	25.8	.002	.48	.002
1/1	.021	.47	.43	.035	25.7	.002	.48	.002
2/1	.082	.80	.45	.034	25.3	.002	.47	.002
5/1	.20	1.40	.46	.039	24.6	.001	.48	.001
7/1	.25	1.73	.45	.040	25.6	.001	.46	.001
10/1	.29	2.14	.48	.049	24.9	.001	.48	.002
15/1	.33	2.27	.47	.050	25.5	.001	.46	.002

With successive stage-wise contacts of solvent with the same feed, at A/O 5/1, the final loaded solvent analyzed, in g/ℓ: 3.0 Cu, 0.025 Fe, 0.011 Zn and 0.001 Pb. No explanation can be given at this time for the difference in copper loading.

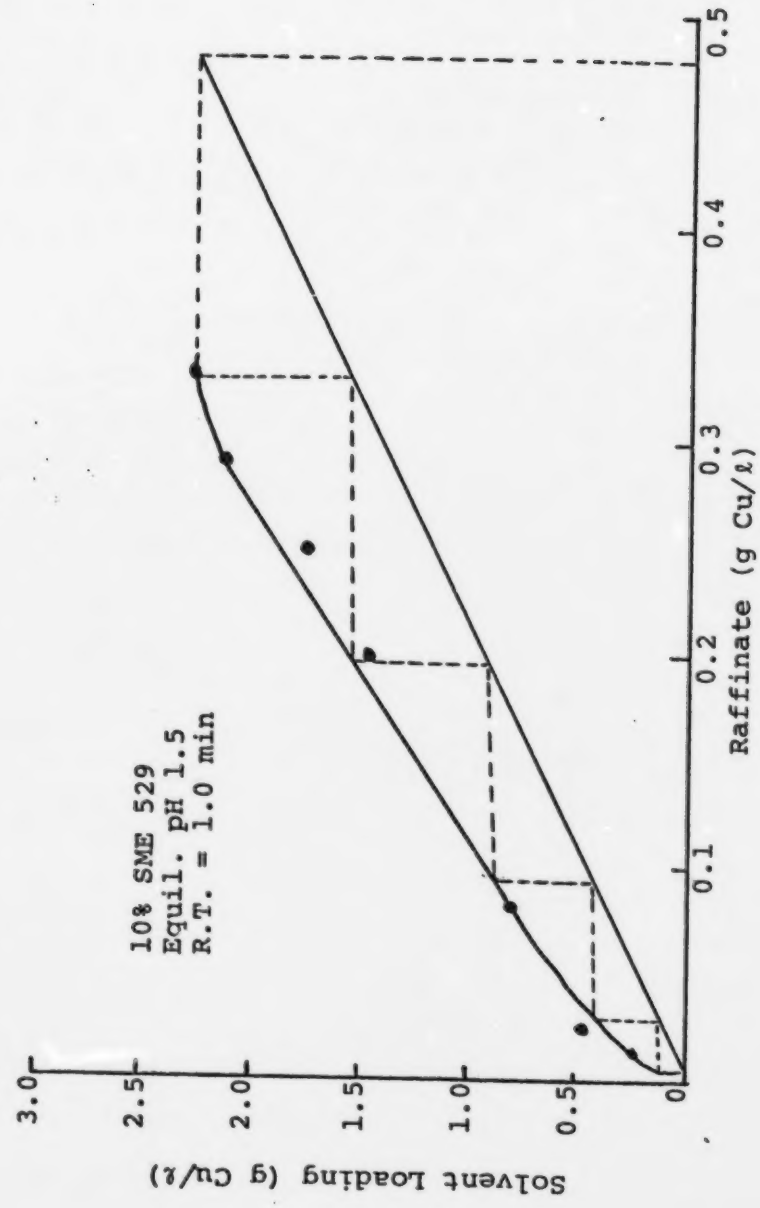

Figure 1. Equilibrium Curve for Cu - SME 529

Zinc Extraction from Copper Raffinate

Screening of Extractants

The summary of screening tests on some selected extractants for zinc are shown in Table 5. The extractants were contacted, at A/O 6, with a feed solution containing, in g/ℓ: 0.5 Fe, 25 Zn, 0.4 Pb. The NaCl was varied from 0 to 3M and the equilibrium pH at 1 and 2. Very little difference was found with change in pH of extraction. The values in Table 5 are shown for extraction at an equilibrium pH 2.0. All solvents were pre-equilibrated with 150 g HCl/ℓ, A/O 5, except where noted otherwise. The diluent for all the solvent mixtures was Shell 140.

The following may be concluded from these screening tests:

(1) Although D2EHPA was an effective extractant for iron, it is well recognized that stripping of iron from that extractant is difficult.

(2) TBP resulted in co-extraction of Fe with Zn and therefore could possibly be used for a two-stage separation. TBP was the best extractant for zinc and the most selective over iron. Also, it was the only reagent which appeared to extract any amount of lead. TOPO was the next best extractant for zinc.

(3) Adogen 383, with 18% Fe extraction was the most selective reagent for iron, (next to D2EHPA). This reagent, at 10% strength, co-extracted 1% zinc compared to 75% TBP where 25% iron was extracted together with 8% zinc. TOPO was the next most selective for iron.

TABLE 5

Screening Tests for Extraction of Zn from Cu Raffinate

Extractant	Loaded Solvent (g/ℓ) NaCl (M) 0			1.0			3.0			Zn/Fe Ratio in Solvent
	Fe	Zn	Pb	Fe	Zn	Pb	Fe	Zn	Pb	
5% LA 3	.002	.045	.005	.002	.11	.011	.002	.14	.006	-
5% Adogen 368	.005	1.90	.006	.012	2.55	.003	.13	2.60	.002	-
5% Adogen 383	.049	1.93	.002	.14	1.99	.002	.55 (18)	1.81 (1.2)	.001	3.3
5% Adogen 464	.003	2.90	.010	.008	2.95	.005	.14	3.28	.002	-
5% D2EHPA	1.87	1.94	.002	1.95	2.02	.001	2.42 (80.6)	1.49 (1)	.001	0.62
5% TOPO	.15	3.18	.003	.13	3.23	.002	.39 (13)	3.18 (2.1)	.001	8.2
5% TOPO (not equil.)	.10	2.90	.003	.17	3.23	.002	0.35	3.18	.002	-
100% TBP	.005	13.1 (8.7)	.027	.07	23.4 (15.6)	.017	1.16 (52)	27.2 (18.1)	.010	23.4
75% TBP	.006	7.64 (5.1)	.017	.032	15.1 (10.1)	.012	0.74 (24.7)	17.6 (11.7)	.006	23.8

( ) indicates % extraction

(4) An increase in extraction was achieved with increase in NaCl concentration, but no effect was shown for change in pH from 1-2.

Extraction of Zinc with TBP

As a result of the screening tests in the previous table, 100% TBP was selected for Zn extraction studies. Solvent was acid equilibrated with 150 g HCl/ℓ, A/O 5/1 before use. Contact time was 5 minutes at A/O 1. Extraction was varied at an equilibrium pH 1-5 on a solution containing, in g/ℓ: 0.5 Fe, 0.39 Pb, 25.2 Zn. The NaCl content was varied 0-3M. The results indicated little increase in extraction of zinc by either increase in equilibrium pH or increase in NaCl concentration. The best phase disengagements were generally at pH 4 and 5. Also there was an improvement with increase in the NaCl concentration, however the phase disengagement was not good.

A similar series of tests was repeated using 75% TBP in Shell 140. Essentially the same results as above were obtained but with lower Zn extraction. Again, phase disengagement was poor. Subsequently, the diluent was changed to Solvesso 150, resulting in much better phase separation over the pH and NaCl concentration ranges tested. The results are shown in Table 6. The feed solution contained, in g/ℓ: 23.9 Zn, 0.48 Fe, 0.40 Pb, and the tests were at an A/O of 0.75. The test results showed a significant increase in the loading (extraction) of zinc with an increase in the chloride concentration. The extraction of lead tended to decrease as the chloride content was increased

TABLE 6

Effect of NaCl Concentration and Equilibrium pH on the Extraction With 75% TBP in Solvesso 150

NaCl Added (M)	Equil. pH	Loaded Solvent (g/ℓ)			Extraction (%)		
		Zn	Fe	Pb	Zn	Fe	Pb
0	1.0	5.94	.002	.014	33.0	0.55	4.6
	2.0	5.84	.002	.012	33.8	.55	4.0
	3.0	6.28	.002	.014	37.6	.55	4.6
	4.0	6.34	.004	.012	39.7	1.11	4.0
	5.0	6.31	.001	.008	35.1	.22	2.7
1.0	1.0	9.44	.018	.012	52.5	4.9	4.0
	2.0	9.44	.007	.012	52.5	1.9	4.0
	3.0	9.66	.003	.012	53.8	.83	4.0
	4.0	9.78	.003	.011	54.4	.83	3.7
	5.0	9.69	.002	.010	53.8	.55	3.3
3.0	1.0	9.88	.24	.006	55.0	66.5	2.0
	2.0	9.63	.043	.006	53.6	11.9	2.0
	3.0	9.94	.004	.006	55.3	1.11	2.0
	4.0	9.69	.002	.006	53.9	0.55	2.0
	5.0	10.1	.005	.005	56.2	1.4	1.7

from 0-3M NaCl. The iron results tended towards increased extraction as the chloride concentration was increased. Change of equilibrium pH had no apparent effect on the extraction of any of the three metals.

Effect of TBP Concentration

The TBP concentration was varied at 20, 30, 40, 50 and 60% in Solvesso 150 diluent, and the equilibrium pH was adjusted at pH 1.0 and 2.0. Extraction tests were with a feed solution containing 25.1 g Zn/ℓ, 0.45 g Fe/ℓ, 0.39 g Pb/ℓ and 1.0M NaCl. As the solvent concentration was increased, the

A/O ratio was equally changed to compensate for the extra sites available. The results of the tests again indicated no effect on extraction due to change in pH. The results of the tests, at equilibrium pH 2, are shown in Table 7. Although the zinc extraction, and therefore loading, was increased by increase in solvent concentration, that of iron and lead remained relatively constant. Thus the discrimination of Zn over Fe and Pb was increased with increase in TBP concentration. This is demonstrated in Figure 2. The results indicate maximum selectivity of Zn over Fe and Pb at a TBP concentration of 40-50%, beyond which, higher solvent concentration results in a decrease in metals selectivity.

Lead Extraction

Screening of Extractants

A feed solution containing 4 g Pb/ℓ and 3M NaCl was contacted with four selected amine extractants to determine metal extraction over the pH range 0.5 to 1.5. The extractants, at 10% concentration and containing 10% isodecanol were dissolved in Shell 140. Prior to extraction, at A/O 1 for 5 minutes, the solvents were pre-equilibrated with 150 g HCl/ℓ at A/O 5. The results in Table 8 indicate only the quaternary amine, Adogen 464, to offer a slight possibility as an extractant for lead. Only a slight enhancement of extraction is obtained by increase in the equilibrium pH from 0.5 to 1.5.

TABLE 7

Effect of Solvent Concentration on Extraction of Zinc and Discriminations over Iron and Lead

Solvent Conc. v/o TBP	A/O Ratio	Loaded Solvent (g/ℓ)					Extraction (%)		
		Zn	Fe	Pb	Zn/Fe	Zn/Pb	Zn	Fe	Pb
20	.2/1	.69	.001	.002	690	345	13.7	1.09	2.56
30	.3/1	1.58	.002	.003	790	527	21.0	1.43	1.02
40	.4/1	2.91	.0007	.004	4157	727	29.0	0.38	2.56
50	.5/1	4.88	.001	.006	4880	813	38.9	0.43	3.07
60	.6/1	6.50	.003	.008	2166	812	43.0	1.08	3.40
75	.75/1	9.44	.007	.012	1348	787	52.5	1.9	4.0

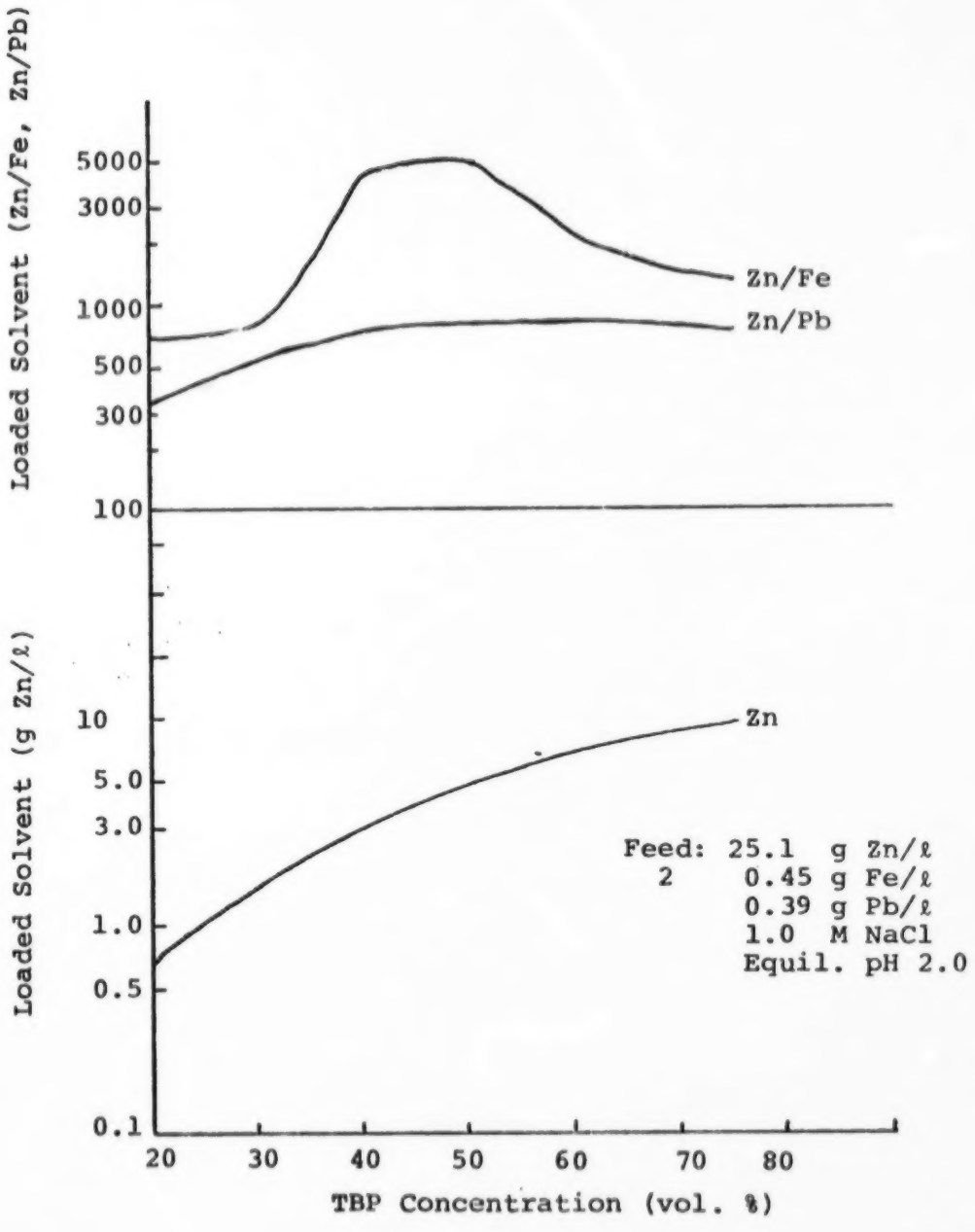

Figure 2. Effect of TBP Concentration on Zn Extraction and Discrimination Over Fe and Pb.

TABLE 8

Screening of Amines for Extraction of Lead

Feed 4.0 g Pb/ℓ, 3M NaCl

Extractant	pH Feed	pH Equil.	Raffinate Pb (g/ℓ)	Loading Calc. (g Pb/ℓ)	% Ext'n Pb
Primene JMT	0.5	0.59	3.97	0.03	
Amberlite LA2		0.60	3.96	0.04	
Alamine 336		0.56	3.74	0.26	
Adogen 464		0.52	2.53	1.47	36.7
Primene JMT	1.0	1.1	3.87	0.13	
Amberlite LA2		1.07	3.96	0.04	
Alamine 336		0.99	3.63	0.37	
Adogen 464		0.49	2.59	1.41	35.2
Primene JMT	1.5*	1.53	3.84	0.16	
Amberlite LA2		1.51	3.93	0.07	
Alamine 336		1.51	3.57	0.43	
Adogen 464		1.50	2.39	1.61	40.2

* This series maintained at pH 1.5

DISCUSSION

Although the tests described here on the evaluation of various extractants for selective extraction of metals from chloride solutions have indicated many possible routes, still more work is required.

If we assume at this time a low iron content in the feed liquor from the chlorination-roast-leach, where no separation process for that metal is required, then the alternatives for the copper and subsequent zinc extraction have been narrowed. Also, although the work was over a range

of NaCl concentration (to provide a chloride background effect for enhancing the metals separation), it is very likely that future work will be directed towards solutions with 3M NaCl to fit closely with the brine leach for lead recovery.

The previous work[1] as well as the initial work in the present investigation indicated SME 529 to be the most suitable extractant for copper extraction. However, subsequent work has indicated that Acorga P5100 or P5300 surpass the extraction characteristics of the SME 529. Therefore, kinetic and equilibrium studies for extraction and stripping will be required on the two new extractants before the final selection of the copper extractant is made prior to small-scale continuous testing.

For zinc, the best extractant appears to be TBP, both for extraction and metal selectivity. Also the use of that reagent provides a system whereby the zinc can be water-stripped to produce a $ZnCl_2$ solution for electrolysis and regeneration of chlorine to roasting.

Only preliminary tests have been done regarding the extraction of lead. The main reason for this is that the front-end of the process is not yet established. If lead can be concentrated in the leach solution, followed by cooling and crystallization, the supernatent returned to leaching will carry only that quantity of lead not crystallized and a solvent extraction process may not be necessary. If there is a requirement to remove lead from the zinc raffinate, the tests to date on a 3M NaCl liquor have indicated that the quaternary

amine Adogen 464 is the best extractant. Obviously, further work is necessary to screen many other possible extractants. Also, the task will be simplified once the leach-crystallization process conditions have been more clearly established.

CONCLUSIONS

To date, the following sequence of extraction and recovery appears possible for stagewise separation and recovery of copper, zinc and lead from chloride solution containing 3M NaCl and in the pH range 1-4:

- Copper extracted with one of the reagents of Acorga P5100, P5300 or SME 529, stripped with copper sulphate return electrolyte, and electrowon to produce cathode copper;
- Zinc extracted with TBP, and water-stripped to produce $ZnCl_2$ which would go to fused salt electrolysis or electrowinning to produce cathode zinc and chlorine for recycle to chlorination;
- Lead would be leached in the brine leach operation, solution cooled to crystallize the $PbCl_2$ for subsequent electrolysis to metal and chlorine recycle, with the remaining lead in the supernatent recycled back to leaching. Alternatively, if it is necessary to extract lead, Adogen 464 is a possible extractant and lead could possibly be recovered by water-stripping to produce a $PbCl_2$ solution.

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